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PATENT SPECIFICATION

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NO DRAWINGS

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Inventor: ALAN JAMES PERKINS

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COMPLETE SPECIFICATION

Improved Carbonized or Graphitized Rayon

We, GREAT LAKES CARBON CORPORATION, a corporation organized under the laws of the State of Delaware, United States of America, of 18 East 48th Street, New York, State of New York, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the production of carbonized or graphitized fibrous rayon materials having improved properties.

It is well known in the art that rayon fibrous materials, such as rayon filaments or rayon textiles or woven fabrics, may be carbonized or graphitized by heating the starting rayon material in an inert atmosphere to suitable temperatures, for example to 800° C. (or to temperatures between about 500° C. and about 1200° C.) to bake or carbonize same, or to 2000°—3000° C. to graphitize same.

The prior art also teaches or suggests some different temperature-time conditions or rates in order to carbonize or graphitize rayon. United States Patents 3,011,981 to Soltes and 3,053,775 to Abbott and British Patent 894,458 may be said to be representative of various conditions or rate schedules for carbonizing or graphitizing rayon fibrous materials.

This invention, as previously stated, also relates to the carbonization or graphitization of rayon, but it is most particularly related to a particular step in such processes, which step leads to carbonized or graphitized products of improved quality as compared to the products produced when the step is not carried out, or greatly shortened heating cycles to obtain products of equal or improved quality.

It is a finding of this invention that if rayon is heated to a temperature within the range of about 325° C. to about 390° C. and more preferably about 340° C. to about 360° C., and is then thoroughly flexed or ruffled or mechanically worked after it has been heated to this temperature and before heating it further, then this procedure, viz. interrupting the heating of the rayon at a given point and flexing same, results in end carbonized or graphitized products having properties which are considerably improved over the properties the products have if this step is not carried out, but identical heating procedures and times are otherwise employed; or in greatly decreased heating schedules or processing times to obtain products whose properties are as good as or better than those obtainable only by greatly prolonging the heating cycles.

It is important, to achieve these improvements, that the flexing be carried out after the rayon has been heated only to a certain temperature point, not too low nor too high. For example, if the ruffling is carried out after the rayon has been heated to a temperature of only about 310° C. or lower, the property improvements and processing advantages brought about by ruffling are not realized; nor are they brought about if the rayon has first been heated to too high a temperature, e.g., 410° C. or higher, before it is ruffled or thoroughly flexed. However, the property improvements are generally obtainable in some measure if the step is carried out and the heating interrupted anywhere in the range between about 325° C. and about 390° C. Carrying out of the step after the rayon has been heated to a temperature in the range of about 340° C. to about 360° C. invariably results in some improvement in the properties and these improvements are generally very marked.

The property improvements being referred to as achievable by the procedures of this invention are improved flexibility and tensile strength of the final carbonized or graphitized

[*Price 4s. 6d.*]

products which is not to say the other property improvements are not also obtained by the procedures involved herein.

The following examples set forth in Tabular form, further illustrate the invention and contrast it with prior art procedures and results. Examples 1 to 4 are illustrative of the invention and Examples 5 to 10 are comparative examples only.

TABLE I

Ex.	Starting Material	Heating Procedure	Flexing Step	Properties of Carbonized Product (Baked to 800° C.)	Properties of Graphitized Product (Graphitized to 2800° C.)
1	Viscose rayon filaments	Room temp. to 350°C.— 15 min. 350°C. to 800°C. — 10 min. 800°C. to 2800°C. — 10 min.	Flexing carried out by thorough "hand working" until the material was soft and supple after the rayon was heated to 350°C. and cooled to <40°C.	About 0.3 gram/denier tensile strength Excellent flexibility	About 0.4 gram/denier tensile strength Excellent flexibility
2	Viscose rayon cloth	Same as in Example 1	Same as in Example 1	About 0.3 gram/denier tensile strength Excellent flexibility	About 0.4 gram/denier tensile strength Excellent flexibility
3	Saponified acetate rayon filaments	Same as in Example 1	Same as in Example 1	About 0.4 gram/denier tensile strength Excellent flexibility	About 0.5 gram/denier tensile strength Excellent flexibility
4	Saponified acetate rayon cloth	Same as in Example 1	Same as in Example 1	About 0.4 gram/denier tensile strength Excellent flexibility	About 0.5 gram/denier tensile strength Excellent flexibility
5	Same as in Example 1	Same as in Example 1	*No* intermediate flexing step	<0.1 gram/denier tensile strength Flexible	<0.1 gram/denier tensile strength Flexible
6	Same as in Example 2	Same as in Example 2	*No* intermediate flexing step	<0.1 gram/denier tensile strength Flexible	<0.1 gram/denier tensile strength Flexible

TABLE I (Continued)

Ex.	Starting Material	Heating Procedure	Flexing Step	Properties of Carbonized Product (Baked to 800 °C.)	Properties of Graphitized Product (Graphitized to 2800 °C.)
7	Same as in Example 2	Room temp. to 310 °C. ~ 15 min. 310 °C. to 800 °C. ~ 10 min. 800 °C. to 2800 °C. — 10 min.	Flexing carried out after the rayon was heated to 310 °C. and cooled to <40 °C.	<0.1 gram/denier tensile strength Flexible	<0.1 gram/denier tensile strength Flexible
8	Same as in Example 2	Room temp. to 410 °C. ~ 15 min. 410 °C. to 800 °C. ~ 10 min. 800 °C. to 2800 °C. — 10 min.	Flexing carried out after the rayon was heated to 410 °C. and cooled to <40 °C.	<0.1 gram/denier tensile strength Flexible	<0.1 gram/denier tensile strength Flexible
9	Same as in Example 2	Room temp. to 350 °C. ~ 30 min. 350 °C. to 800 °C. — 10 min. 800 °C. to 2800 °C. — 10 min.	No intermediate flexing step	<0.1 gram/denier tensile strength Flexible	<0.1 gram/denier tensile strength Flexible
10	Same as in Example 2	Room temp. to 350 °C. ~ 35 hrs. 350 °C. to 400 °C. ~ 5 hrs. 400 °C. to 800 °C. ~ 8 hrs. 800 °C. to 2800 °C. ~ 2 hrs.	No intermediate flexing step	About 0.3 gram/denier tensile strength Excellent flexibility	About 0.4 gram/denier tensile strength Excellent flexibility

It will be noted from the foregoing Table, particularly Examples 1—4, that carbonized and graphitized products having very good properties are obtainable by the processes of this invention and regardless of whether the starting rayon material is of the viscose type, or the saponified acetate type, and also whether the rayon is in filamentary or woven fabric or cloth form, etc. It will also be noted from these same Examples that the heating rates which may be employed in the present invention, with the obtainment of very good properties, may typically be as follows:

Room temperature to 350° C.— about 1000°— 1300° C./hr.
350° C. to 800° C.— about 2500°— 2700° C./hr.
800° C. to 2800° C.— about 10000°—12000° C./hr.

It will also be noted that the starting materials and processing conditions in Examples 5 and 6 are exactly the same as in Examples 1 and 2 except for the omission of the intermediate flexing step of this invention. The advantages and product improvements realizable because of the step of this invention are readily apparent.

Examples 7 and 8 demonstrates the critical nature of the temperature range to which

the cloth must be heated before it is flexed. If the cloth is heated to too low a temperature and then flexed, as in Example 7, or heated to too high a temperature and then flexed, as in Example 8, the product improvements are not obtainable, even though substantially identical heating schedules and processing conditions are otherwise followed.

Example 9 demonstrates the fact that even though much slower heating rates to 350° C. are employed than in Example 1, because of the omission of the flexing step, the product qualities obtained are still not as good as those obtained in Example 1.

Example 10 demonstrates that product properties comparable to those obtained in Example 1 are obtainable without employing the flexing step, but that very greatly prolonged heating rates or times must be employed in order to accomplish this.

If the rayon being processed is in filamentary form or if its width is of relatively small dimension then flexing in substantially only one direction, the longitudinal of the material, is generally all that is necessary. However, if the rayon is in the form of a woven fabric or cloth, having both warp and fill filaments, or is of substantial width, then flexing in both longitudinal and transverse directions are necessary to achieve optimum results and to insure the obtainment of a carbonized or graphitized product strong or flexible in one direction, and not noticeably weaker or less flexible, in the other direction.

By "flexing" is meant working or ruffling or pressing the rayon, after it has been heated to a suitable temperature, until it is soft or supple or very flexible. (The heat-treated rayon at this point, before being flexed, is generally fairly stiff or rigid and also coarse to the touch rather than soft.) A typical way of flexing the cloth can be accomplished by the use of standard commercially available calendar rolls, such as used in textile operations, with the only limiting factor being the pressure applied by the rollers, such that the cloth is not physically damaged; but sufficient pressure is employed to render the cloth supple and flexible.

For convenience in handling the rayon which has been heated to a suitable temperature, will typically be cooled to temperatures below about 40° C. or to room temperatures before the flexing stop is carried out; however, that is not absolutely essential to the invention.

After the flexing step has been carried out, the heat-treated and flexed rayon may be heated substantially immediately or over a very short time period to the temperature it had previously been heated to before it was flexed; thereafter, it may be heated to carbonizing or graphitizing temperatures following any convenient time-temperature schedules, such as outlined in the Examples.

The flexing can be accomplished by hand simply by working the product between the palms of one or more persons until the "hand" or "feel" of the material is proper; or it can be accomplished by passing the heat-treated rayon between two opposed rollers one or more times until sufficient flexibility is effected. Rubber or plastic coated rollers are preferred—either one rubber, etc., with one steel or metal roller, or both rubber or plastic coated.

It should be understood that slower heating rates, than those previously pointed out as typical, may also be employed. However, rates slower than 200° C. per hour, in going from room temperature to the 325—390° C. range, or in going to the further carbonization temperature (which is exemplified as 800° C., but which typically may be anywhere between about 500° C. and about 1200° C.) will seldom be employed because inexpedient and because of the prolonged processing times involved.

WHAT WE CLAIM IS:—

1. A process of carbonizing fibrous rayon which comprises heating the rayon under substantially non-oxidizing conditions to a temperature between 325° C. and about 390° C.; interrupting the heating step at a temperature in this range and flexing the rayon by mechanically or manually working the product to make it more supple; and continuing the heating of the rayon under non-oxidizing conditions to a temperature above about 500° C. to further the carbonization thereof.

2. A process according to claim 1 wherein the rayon is heated from room temperature to the 325° C.—390° C. range at a rate no slower than 200° C. per hour, and wherein the flexed material is then further carbonized by heating at a rate no slower than 200° C. per hour.

3. A process according to claim 1 or 2 wherein the flexing of the rayon is carried out after it has been cooled to a temperature no higher than about 40° C.

4. A process according to claim 1, 2 or 3 wherein the final temperature to which the rayon is heated is between about 500° C. and about 1200° C.

5. A process according to claim 1, 2 or 3 wherein the final temperature to which the rayon is heated is between about 2000° C. and about 3000° C.

6. A process according to any of the preceding claims wherein the initial temperature to which the rayon is heated before it is flexed is between about 340° C. and about 360° C.

7. A process according to any of the preceding claims wherein the rayon is viscose rayon.

8. A process according to any of the pre-

ceding claims wherein the rayon is in the form of a woven fabric.

9. A process according to claim 8 wherein the flexing of the woven fabric is carried out in directions both longitudinal and transverse to the fabric.

10. A process of carbonizing rayon as claimed in claim 1 substantially as herein described with particular reference to the accompanying examples 1 to 4.

STEVENS, LANGNER, PARRY & ROLLINSON,
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Agents for the Applicants.

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